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### (54) POLYGLYCERIN FRACTIONAL MATTER, ITS FATTY ACID ESTER AND ITS PRODUCTION

(57)Abstract:

PURPOSE: To produce a surfactant capable of producing a completely solubilized matter and a stably emulsified matter in food, cosmetics, pharmaceuticals and industrial field.

CONSTITUTION: A polyglycerin ester prepared by the esterification of polyglycerin, obtained by removing low-molecular-weight polymerized matter from a polyglycerin reaction product, with fatty acid is suitable for the production of the completely solubilized matter and the stably emulsified matter and the production of a product impossible higher to becomes possible.

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CLAIMS

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[Claim(s)]

[Claim 1] The fatty acid ester obtained according to esterification with the polyglycerin fraction which removed the low-molecular reactant of a polyglycerin reactant and was obtained, and the fatty acid following it, and its manufacture approach.

[Claim 2] The polyglycerin fraction according to claim 1 characterized by decreasing a with a polymerization degree of two or less low-molecular reactant to 10% or less, its fatty acid ester, and its manufacturing method.

[Claim 3] The polyglycerin fraction according to claim 1 characterized by decreasing a with a polymerization degree of three or less low-molecular reactant to 30% or less, its fatty acid ester, and its manufacturing method.

[Claim 4] The polyglycerin fraction according to claim 1 to 3 characterized by removing a low-molecular reactant by molecular distillation, its fatty acid ester, and its manufacture approach.

[Claim 5] The polyglycerin fraction according to claim 1 to 3 characterized by removing a low-molecular reactant by the vacuum distillation which uses a steam as a carrier, its fatty acid ester, and its manufacture approach.

[Claim 6] The polyglycerin fraction according to claim 1 to 3 characterized by removing a low-molecular reactant with false moving-bed mold liquid chromatography, its fatty acid ester, and its manufacture approach.

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Industrial Application] The polyglyceryl fatty acid ester obtained by this invention can be used about the manufacture approach of a polyglycerin fraction of using this invention as the high polyglyceryl fatty acid ester of the capacity to reduce surface tension, and its raw material, as a surfactant of a food additive, the object for cosmetics, the object for physic, and industrial use for the purpose, such as emulsification, solubilization, distribution, washing, corrosion prevention, lubrication, electrification prevention, and wetting.

[0002]

[Description of the Prior Art] Food-grade surfactants, such as a nonionic surface active agent of ethylene oxide systems, such as various compounds, for example, polyoxyethylene alkyl ether, polyoxyethylene polyhydric-alcohol fatty acid ester, and polyoxyethylene alkyl phenyl ether, a sorbitan fatty acid ester, sucrose fatty acid ester, and polyglyceryl fatty acid ester (polyglycerin condensation ricinoleic-acid ester is included), are conventionally known as emulsification or a solubilizing agent. Also in it, polyglyceryl fatty acid ester can acquire the safety to the body and an environment, and the presentation of varieties, and since versatility is high, it is the most useful surfactant. These polyglyceryl fatty acid ester and polyglycerin condensation ricinoleic-acid ester were manufactured by carrying out an esterification reaction, using as a raw material the polyglycerin reactant and fatty acid which carried out the polymerization of the glycerol at the elevated temperature under existence of alkali catalysts, such as caustic alkali of sodium, and were decolorized [ were deodorized and ] and obtained as one of the raw materials. Or the reactant obtained by carrying out and carrying out chemosynthesis of epichlorohydrin, glycidol, a glycerol or polyglycerin, epichlorohydrin, a mono-chlorohydrin, the dichlorohydrin, or the glycidol to a raw material again was used, having refined remaining as it is or if needed. these polyglycerin reactant -- the purpose of deodorization or removal of an unreacted raw material -- the bottom of heating, and several -- on the reduced pressure conditions of Torr, using adsorbents, such as activated carbon, a color component and a smell component be remove, or reduction processing be carry out by hydrogenation etc. again, and it be refine [ \*\*\*\* / and / remove ion components, such as a catalyst which used it by ion exchange resin, ion exchange membrane, etc., ]. [ lead gases, such as nitrogen and a steam, ] However, processing of these polyglycerin reactant was what does not do effect at all about presentation distribution of a polyglycerin reactant, respectively.

[0003]

[Problem(s) to be Solved by the Invention] The polyglycerin which is circulating in the commercial scene is called the tetra-glycerol, the hexa glycerol, and the deca glycerol by the degree of polymerization of the average calculated from the hydroxyl value. However, it is the mixture of the various glycerol polymerization objects from polymerization degree 1 to ten or more in fact. Generally, in these polyglycerin and a fatty acid, when compounding ester by the esterification reaction, the reactivity differs and the polyglycerin of low polymerization degree is alternatively esterified by the polymerization degree of a glycerol. Consequently, the resultant acquired turns into mixture of the

polyglyceryl fatty acid ester with an ester bond with many fatty acids of low polymerization degree, and unreacted high-polymer polyglycerin without an ester bond rather than it means. Therefore, they were the emulsification which it originally has, and the thing which cannot demonstrate the solubilization force. For example, when useful matter, such as a vitamin of lipophilicity, such as vitamin E, and carotene, was added and manufactured to a drink, in the existing food-grade surfactant, transperence solubilization could not be carried out and a product with sufficient preservation stability was not able to be manufactured. Moreover, in case drugs and cosmetics are manufactured, other surface active agents, for example, polyoxyethylene sorbitan ester, it can be used, but since there is not sufficient solubilization force if independent when adding and manufacturing useful matter, such as a vitamin of lipophilicity, such as vitamin E, and carotene, to a drink, assistants, such as ethanol, are required. Therefore, if it drinks in a large quantity, it will be in an inebriation condition and, especially in the case of the youth, has become a social problem. Furthermore, although the polyoxyethylene derivative is used as an emulsifier of a hydrophilic property in the cosmetics industry, a problem is in safeties, such as skin irritation, and the substitute is called for. However, conventional polyglyceryl fatty acid ester and sucrose fatty acid ester of engine performance are inadequate, and cannot be substituted. Although the approach of manufacturing high-definition polyglyceryl fatty acid ester using a metal oxide catalyst is indicated in JP,4-145046,A, although an esterolysis and coloring improve, since especially the molecular weight distribution of polyglycerin are uncontrollable by this approach, surface activity capacity of ester [ especially ] obtained does not improve. Although the approach of using and manufacturing lipase is indicated in JP,61-187795,A, JP,61-257191,A, JP,61-257191,A, and JP,3-151885,A in case polyglycerin is esterified, surface activity capacity of this approach does not improve. In JP,63-23837,A and JP,3-81252,A, after esterifying, it has tried to solvent judgment will remove unreacted polyglycerin and improve surface activity capacity, but by this approach, a process becomes complicated, and since yield is low, practical use cannot be presented.

[0004]

[Means for Solving the Problem] It was hyperviscosity as that the industrial semantics of removing the low-molecular reactant of a polyglycerin reactant conventionally is not recognized, and a physical property of polyglycerin, and especially since the boiling point was high and handling was difficult, it did not inquire. Therefore, it found out that it could manufacture by performing the vacuum distillation or liquid chromatography which makes molecular distillation and a steam a carrier for removing the low-molecular reactant in a polyglycerin reactant as a result of inquiring wholeheartedly. Furthermore, having the outstanding surface activity capacity also finds out the polyglyceryl fatty acid ester obtained considering these polyglycerin fraction as a raw material, and it came to complete this invention. Namely, this invention relates to the polyglyceryl fatty acid ester obtained according to esterification with the polyglycerin fraction which removed the low-molecular reactant in a polyglycerin reactant, and was obtained, and the fatty acid following it, and its manufacture approach. Decrease preferably a with a polymerization degree of two or less low-molecular reactant to 1% or less 10% or less in detail. Or it is related with the fatty acid ester obtained according to esterification with the polyglycerin fraction which decreased preferably the with a polymerization degree of three or less low-molecular reactant to 1% or less 30% or less, and the fatty acid following it, and its manufacture approach.

[0005] This invention is explained to a detail below. In case the surfactant of this invention mixes the oleophilic matter and the hydrophilic matter, it is the matter added for the purpose of stabilization, and it has strong surface activity ability. These matter has both an oleophilic functional group and the functional group of a hydrophilic property in the intramolecular, and has the capacity to reduce boundary tension and surface tension. The polyglycerin of this invention is with a polymerization degree of two or more acquired by carrying out dehydration condensation of the glycerol polyglycerin, is matter which has a hydroxyl group and ether linkage in intramolecular, and does not have other functional groups, and points out all the things that have equivalent structure regardless of how of a raw material process. The polyglycerin reactant of this invention usually heats a glycerol under ordinary pressure or reduced pressure under an alkali catalyst, and is obtained. Or a polyglycerin reactant can be obtained even if it compounds and refines them, using glycidol, epichlorohydrin, a mono-chlorohydrin, etc. as a

raw material. Furthermore, a glycerol or the partial alcoholate of the polymer, and halogenated hydrocarbon or oxy-halogenated hydrocarbon can be used as a raw material, and can be obtained by the dehalogenation alkali-metal salt reaction. These polyglycerin reactant contains many raw materials, such as an unreacted glycerol, polyglycerin of polymerization degree 2, and polyglycerin (low-molecular reactant of this invention) of polymerization degree 3. moreover, these polyglycerin reactant -- the purpose of deodorization or removal of an unreacted raw material -- the bottom of heating, and several - on the reduced pressure conditions of Torr, using adsorbents, such as activated carbon, a color component and a smell component may be remove, or reduction processing may be carry out by hydrogenation etc. again, and you may refine [ \*\*\*\* / and / remove ion components, such as a catalyst which used it by ion exchange resin, ion exchange membrane, etc., ]. [ lead gases such as nitrogen and a steam, ]

[0006] Conventionally, if dehydration condensation of the glycerol is carried out, paying attention to two mols of hydroxyl groups of intramolecular changing into one mol of ether linkage, the polymerization degree of polyglycerin would measure the hydroxyl value by the criteria fats and fatty oils test, and will be understood as what calculated the polymerization degree of the average. In case low-molecular to a macromolecule had large molecular weight distribution and the polyglycerin with which this is conventionally supplied to the commercial scene expressed a presentation numerically, it was complicated, and as this invention described, it was because there was no knowledge which observes molecular weight distribution and the surface activity capacity of the ester obtained. However, in having used the measuring method of such polymerization degree, when considering the property of the polyglyceryl fatty acid ester obtained from these polyglycerin, it is unsuitable. Therefore, the degree of polymerization of the polyglycerin in this invention shall perform a separation quantum for polyglycerin, such as trimethylsilylation or acetylation, by the GC method (gas chromatography) a derivative, nothing, and on it, and shall calculate it in an area method. Analysis by the GC method can be easily carried out, if temperature up analysis for 10-degree-C/is performed to 100 degrees C - 250 degrees C using fuze DOSHIRIKA capillary tube tubing to which the chemical bond of the low polar liquid phase, such as methyl silicone, is carried out. Moreover, identification of the peak on gas chromatogram can introduce a gas chromatograph into the double-focusing mass spectrograph, it can be ionized by approaches, such as chemical eye demon ZESHON, and it can measure it, and can be easily performed by calculating the molecular weight of the peak on gas chromatogram from the molecular weight of the parent ion, and next, searching for the polymerization degree of a glycerol from a chemical formula further. The polyglycerin fraction of this invention is polyglycerin which made the low-molecular reactant in the polyglycerin reactant obtained by performing molecular distillation, the vacuum distillation which makes a steam a carrier, or liquid chromatography remove or decrease. It is polyglycerin which decreased with a polymerization degree of two or less polyglycerin to 1% or less preferably 10% or less since the one where molecular weight distribution are narrower is desirable as this polyglycerin fraction was mentioned above, a bad influence with the big polyglycerin of especially low polymerization degree was done and it was necessary to lessen especially, or decreased with a polymerization degree of three or less polyglycerin to 3% or less preferably 30% or less.

[0007] The molecular distillation of this invention uses the bottom of a high vacuum for the distance of 25cm or less 50cm or less 100cm or less near the heating surface in the equipment which installed the condensation plane still more preferably preferably so that a raw material may be extended in the shape of a thin film with a centrifugal force, a brush, or a roll on the heating surface and the molecules which evaporated may be hard to collide, it uses the difference of the vapor pressure of the matter on condition that an elevated temperature, and is separation and a technique which carries out fractionation. On the conditions of the molecular distillation of this invention, i.e., the degree of vacuum of 0.01 - 2torr, it is the degree of vacuum of 0.01 - 1torr preferably, is the degree of vacuum of 0.01 - 0.5torr still more preferably, is a 150 degrees C - 300 degrees C elevated temperature, and is a 180 degrees C - 280 degrees C elevated temperature preferably, and the polyglycerin fraction which removed the low-molecular reactant can be obtained by distilling at a 220 degrees C - 260 degrees C elevated temperature still more preferably. The vacuum distillation which made the steam of this invention the carrier is a

technique which distills under an elevated temperature and a high vacuum using the equipment designed using \*\*\*\*\* type distillation apparatuses, such as well-known vacuum distillation equipment, for example, falling film vacuum distillation equipment, compulsive thin film vacuum distillation equipment, and restoration, etc. so that a raw material and a counterflow might be made to pass a steam. Under the present circumstances, the 10 times [ 0.01 to ] as many range of the water vapor content to be used as this is desirable to the amount of supply of a raw material, and its twice are still more desirable from 0.1 times. 200 degrees C - 350 degrees C are desirable still more desirable, and temperature conditions are 240 degrees C - 300 degrees C. Moreover, 0.01torr(s) - 0.5torr is desirable still more desirable, and reduced pressure conditions are 0.02 - 0.2torr(s).

[0008] The liquid chromatography of this invention may be equipment which is made to pass a raw material and is separated using the difference of the distribution coefficient among two phases of the solid particulate which coated the front face with the solid particulate or solution layer with which the column was filled up, and an outflow solvent, and may be liquid chromatography of what kind of well-known method. For example, to fixed timing, the raw material and outflow solvent of the specified quantity are supplied to continuation or an intermittence target, respectively, and continuation, the false moving-bed mold method extracted intermittently, or its advanced type can use the effluent of the specified quantity for a predetermined column from a predetermined column in fixed timing at the single column method and 2-16 columns which supply the constant rate of a raw material to the column which filled the outflow solvent, and supply an outflow solvent after that. With the solid particulate with which the column used for liquid chromatography was filled up, or the solid particulate which coated the front face with the solution layer, a silica, A solid-state with adsorption activity, such as an alumina, activated carbon, a polyamide, clay, cerite, and Florisil, On the front face, or acid salt, basic salt, olefins, polyolefine, Aromatic series, a halogenide and oxydi propionitrile, and siloxanes The solid-state which coated silicate ester and glycols or carried out the chemical bond, In a styrene frame or an acrylic frame, as a functional group An amide group, an amine radical, A phosphate group, a sulfonic group, a carboxylic-acid radical or its sodium salt, potassium salt, Lithium salt, a calcium salt, magnesium salt, a hydrochloride, a sulfate, phosphate, It is molecular sieving, such as the ion-exchange resin, ZEORATO, a molecular sieve, polystyrene, the poly dextran, polyacrylamide, and agarose, a gel exclusion mold bulking agent, etc. which have a carbonate, a nitrate, an oxalate, acetate, citrate, etc. Moreover, the outflow solvent of this invention is the solvent or solution which is a liquid or supercritical fluid under ordinary pressure or pressurization. They are adjustment or continuation, and the thing changed gradually about salt concentration, hydrogen ion concentration, and a polarity by the need. Carbon dioxide gas, Alcohols, such as water and methanol ethanol propanol isopropanol, The phosphate solution, a citrate solution, etc. can be illustrated to ester, such as ketones, such as an acetone methyl ethyl ketone, and ethyl acetate, methyl acetate, ethyl formate, methyl formate, or the mixture of those, and a pan. It is in a styrene frame here with the ion exchange resin which has the sodium salt of a sulfonic acid, potassium salt, lithium salt, a calcium salt, and magnesium salt using a false moving-bed mold method or its advanced equipment preferably, and it combines, or it is in an acrylic frame with the ion exchange resin which has the sodium salt of a sulfonic acid, potassium salt, lithium salt, a calcium salt, and magnesium salt using water as an outflow solvent, and is a combination using water as an outflow solvent.

[0009] moreover, the purpose of, maintaining a supercritical condition or reducing the viscosity of the liquid in a column -- temperature -- for example, (40-80 degrees C), a pressure (from 150 atmospheric pressures to for example, 400 atmospheric pressures) may be controlled. [ raising separation efficiency ] The fatty acid of this invention is especially the generic name of the matter which contains the carboxylic acid refined and obtained, without hydrolyzing the fats and oils extracted from natural animals and plants, and dissociating, or dissociating as a functional group, and it does not limit. Or you may be the fatty acid which uses petroleum etc. as a raw material, compounds chemically, and is obtained. Or you may be the polymerization fatty acid obtained by carrying out the heating polymerization of the condensation fatty acid obtained by carrying out condensation polymerization of what hydrogenation etc. carried out these fatty acids and returned, and the fatty acid containing a

hydroxyl group, and the fatty acid which has an unsaturated bond. The mixture of oleic acid, isostearic acid, a palmitic acid, a lauric acid, a capric acid, a caprylic acid, a ricinoleic acid, 12-hydroxy stearic acid, a condensation ricinoleic acid, condensation 12-hydroxy stearic acid, a caproic acid, a heptyl acid, a nonylic acid, undecanoic acid, a myristic acid, stearic acid, palmitoleic acid, behenic acid, linolic acid, a linolenic acid, an elaidic acid, 2 ethylhexyl acid, or these fatty acids can be illustrated. What is necessary is to take into consideration the effectiveness of the product for which it asks in selection of these fatty acids, and just to decide suitably. If an environmental problem etc. is taken into consideration and the fatty acid of the natural animals-and-plants origin desires stability with the passage of time preferably again, the fatty acid which does not have a partial saturation double bond two or more will be desirable.

[0010] The polyglycerin fraction and fatty acid of this invention are esterified by the well-known approach. For example, it can esterify under ordinary pressure or reduced pressure under an alkali catalyst, an acid catalyst, or a non-catalyst. Moreover, the charge of a polyglycerin fraction and a fatty acid must be suitably chosen for the purpose of a product. For example, what is necessary is just to make the mol number of fatty acids increase, if it is going to obtain an oleophilic surfactant that what is necessary is to calculate weight and just to teach so that it may become equimolar from the hydroxyl value of a polyglycerin fraction, and the molecular weight of a fatty acid by count, if it is going to obtain the surfactant of a hydrophilic property. The demand on use of a product may refine the obtained polyglyceryl fatty acid ester. What kind of well-known approach is especially sufficient as the approach of purification, and it is not limited. For example, by activated carbon, the activated clay, etc., and using an acid or alkali, it may wash, or molecular distillation may be performed [, and ], using a steam, nitrogen, etc. as carrier gas, and you may refine. [ carrying out adsorption treatment ] [ performing processing under reduced pressure ] Or carrying out separation removal etc. may carry out unreacted polyglycerin etc. using the liquid-liquid distribution, adsorbent, resin, molecular-sieving, loose reverse osmotic membrane, and ultra fill tray SHON film etc.

[0011] Other components can be added to the polyglyceryl fatty acid ester of this invention, and the handling of a product can be made easy. For example, in order to reduce the viscosity of a product, kinds, such as ethanol, propylene glycol, a glycerol, polyglycerin, water, liquid sugar, and fats and oils, or two sorts or more may be added, and you may dissolve or emulsify. Or disintegration of the protein, such as polysaccharide, caseinate, etc., such as a lactose and a dextrin, may be added and carried out. Other surfactants are mixed with the polyglyceryl fatty acid ester of this invention depending on the purpose of use, and it is good also as surfactant pharmaceutical preparation. The surface active agent which can be used Lecithin, such as a soybean lecithin, yolk lecithin, and rapeseed lecithin, Or the partial hydrolysate, caprylic-acid monoglyceride, capric-acid monoglyceride, Lauric-acid monoglyceride, myristic-acid monoglyceride, palmitic-acid monoglyceride, Stearic acid monoglyceride, behenic acid monoglyceride, oleic acid monoglyceride, Monoglyceride, such as elaidic-acid monoglyceride, ricinoleic-acid monoglyceride, and condensation ricinoleic-acid monoglyceride, or such monoglyceride mixture, Or the acetic acid of these monoglyceride, a citric acid, a succinic acid, a malic acid, The organic-acid monoglyceride which is ester with organic acids, such as a tartaric acid, caprylic-acid sorbitan ester, Capric-acid sorbitan ester, lauric-acid sorbitan ester, myristic-acid sorbitan ester, Palmitic-acid sorbitan ester, stearic acid sorbitan ester, Behenic acid sorbitan ester, oleic acid sorbitan ester, elaidic-acid sorbitan ester, Sorbitan fatty acid esters, such as ricinoleic-acid sorbitan ester and condensation ricinoleic-acid sorbitan ester, Caprylic-acid propylene glycol ester, capric-acid propylene glycol ester, Lauric-acid propylene glycol ester, myristic-acid propylene glycol ester, Palmitic-acid propylene glycol ester, stearic acid propylene glycol ester, Behenic acid propylene glycol ester, oleic acid propylene glycol ester, Elaidic-acid propylene glycol ester, ricinoleic-acid propylene glycol ester, Propylene glycol fatty acid ester, such as condensation ricinoleic-acid propylene glycol ester, Caprylic-acid cane-sugar ester, capric-acid cane-sugar ester, lauric-acid cane-sugar ester, Myristic-acid cane-sugar ester, palmitic-acid cane-sugar ester, stearic acid cane-sugar ester, Behenic acid cane-sugar ester, oleic acid cane-sugar ester, elaidic-acid cane-sugar ester, Nonionic surface active agents and amphoteric surface active agents, such as sucrose fatty acid ester, such as ricinoleic-acid cane-sugar ester and



condensation ricinoleic-acid cane-sugar ester, an anionic surface active agent, a cationic surface active agent, etc. can be illustrated. This invention is not limited by these, although an example is shown below and this invention is concretely explained to it.

[0012]

[Example]

By using as a raw material the polyglycerin reactant of the example 1 of a comparison mentioned later example 1, on 0.4torr(s) and 240-degree C conditions, molecular distillation was performed and the polyglycerin fraction was obtained. When it trimethylsilylated and measured about the obtained polyglycerin fraction using the gas chromatograph, it was bigger polyglycerin 3% than polymerization degree 11 polyglycerin 31% of the polymerization degree 4-11 polyglycerin 38% of the polymerization degree 3 polyglycerin 20% of the polymerization degree 2 glycerol 8%. Having put 500g of ricinoleic acids, and 0.8g of sodium hydroxides into the 1l. 4 Thu openings flask, and removing generation water under a nitrogen air current, it reacted at 210 degrees C and the condensation ricinoleic acid was obtained. The acid number of this condensate was 33.8. Having added 60.5g of polyglycerin fractions which carried out molecular distillation here and which were obtained to it, and removing generation water under a nitrogen air current, it reacted at 250 degrees C and polyglycerin condensation ricinoleic-acid ester was obtained.

[0013] 210g [ of polyglycerin fractions ], 90g [ of isostearic acid ], and phosphoric-acid 3 potassium 0.1g obtained in the example 1 was put into the 4 Thu openings flask of 2500ml of examples, and it reacted at 250 degrees C, removing generation water under a nitrogen air current, the 0.3ml phosphoric acid was added after the reaction, and polyglycerin isostearic acid ester was obtained.

Polyglycerin stearic acid ester was obtained from 225.2g of polyglycerin fractions obtained in the example 3 example 1, and 74.2g of stearic acid according to the completely same process as an example 2.

[0014] Polyglycerin oleic acid was obtained from 225.2g of polyglycerin fractions obtained in the example 4 example 1, and 74.2g of oleic acid according to the completely same process as an example 2.

Molecular distillation was performed on 0.4torr(s) and 240-degree C conditions by having used as the raw material the polyglycerin reactant of the example 1 of a comparison mentioned later example 5, and the polyglycerin fraction was obtained on 0.2torr(s) and 255-degree C conditions to the pan. When it trimethylsilylated about the presentation of the obtained polyglycerin fraction and having been measured using the gas chromatograph, it was bigger polyglycerin 4% than polymerization degree 11 polyglycerin 48% of the polymerization degree 4-11 polyglycerin 41% of the polymerization degree 3 polyglycerin 5% of the polymerization degree 2 glycerol 2%.

[0015] Having put 500g of ricinoleic acids, and 0.8g of sodium hydroxides into the 1l. 4 Thu openings flask, and removing generation water under a nitrogen air current, it reacted at 210 degrees C and the condensation ricinoleic acid was obtained. The acid number of this condensate was 33.8. Having added 60.5g of polyglycerin fractions which carried out molecular distillation here and which were obtained to it, and removing generation water under a nitrogen air current, it reacted at 250 degrees C and polyglycerin condensation ricinoleic-acid ester was obtained.

210g [ of polyglycerin fractions ], 90g [ of isostearic acid ], and phosphoric-acid 3 potassium 0.1g obtained in the example 5 was put into the 4 Thu openings flask of 6500ml of examples, and it reacted at 250 degrees C, removing generation water under a nitrogen air current, the 0.3ml phosphoric acid was added after the reaction, and polyglycerin isostearic acid ester was obtained.

Polyglycerin stearic acid ester was obtained from 225.2g of polyglycerin fractions obtained in the example 7 example 5, and 74.2g of stearic acid according to the completely same process as an example 2.

Polyglycerin oleic acid was obtained from 225.2g of polyglycerin fractions obtained in the example 8 example 5, and 74.2g of oleic acid according to the completely same process as an example 2.

[0016] 1mg water vapor content for /performed falling film vacuum distillation at 0.08torr(s) and 270 degrees C by having used as the raw material the polyglycerin reactant of the example 1 of a comparison

mentioned later example 9, and the polyglycerin fraction was obtained. When it trimethylsilylated about the presentation of the obtained polyglycerin fraction and having been measured using the gas chromatograph, it was bigger polyglycerin 4% than polymerization degree 11 polyglycerin 53% of the polymerization degree 4-11 polyglycerin 33% of the polymerization degree 3 polyglycerin 7% of the polymerization degree 2 glycerol 3%. Having put 500g of ricinoleic acids, and 0.8g of sodium hydroxides into the 1l. 4 Thu openings flask, and removing generation water under a nitrogen air current, it reacted at 210 degrees C and the condensation ricinoleic acid was obtained. The acid number of this condensate was 33.8. Having added 60.5g of polyglycerin fractions obtained here, and removing generation water under a nitrogen air current, it reacted at 250 degrees C and polyglycerin condensation ricinoleic-acid ester was obtained.